BOND TESTING FOR EFFECTS OF SILICONE CONTAMINATION

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Introduction

In 2003 ATK Thiokol discovered that the smocks and coveralls worn by its operations personnel for safety and contamination control were themselves contaminated with a silicone defoamer and a silicone oil. As a growing list of items have been identified as having this form of contamination, it was desirable to devise a test method to determine if the contamination level detected could cause subsequent processing concerns. The smocks and coveralls could potentially contact bonding surfaces during processing so the test method focused on dry transfer of the silicone from the clothing to the bonding surface.

Experimental

The silicone defoamer was used in production of the cloth for the smocks and coveralls while the silicone oil was applied to the sewing thread prior to assembly of the garments. To look at the two different sources of silicone, fresh, unwashed garments were cut into sections; half with the threads in the seams exposed and half with only the cloth. A third set of cloth samples were produced using similar cloth that had not been exposed to the defoamer or silicone oil. In general, the silicone defoamer produced roughly 1000 ppm by weight silicone during chemical extraction testing and the threads 15,000 to 30,000 ppm by weight. The type of silicone was not identified but appeared chemically similar to a polydimethylsiloxane.

Tapered double cantilevered beams, TDCBs, were grit blasted and one half of each set exposed to the cloth. The cloth was placed on the TDCB and pulled across it. It is estimated that the heavy and light pressure ranges were 1.5-2.5 and 0.5-1.0 psi, respectively. These pressures represent an operator leaning against a bond surface with hand or elbow and the approximate pressure resulting. One and three passes, each with a new cloth, were also used representing multiple transference events. Figures 1 through 3 show the configuration of the TDCBs and cloth during the exposure. Figure 4 shows how a similar setup may be used for tensile adhesion testing, though this setup was not used for this investigation. Three beams were produced for each cloth, pressure, and pass combination along with a set of uncontaminated control beams.

Once the TDCB surfaces were exposed, they were blown clean with filtered dried air to remove any particulate left behind. The surfaces, both the exposed and the unexposed twin, were then primed with a Gamma- glycidoxypropyltrimethoxysilane. Bonding was accomplished using one of the most contamination sensitive epoxy adhesives used on the Space Shuttle Reusable Solid Rocket Motors.

Results and Discussion

Table 1 shows the thirteen samples groups and the exposure conditions for each set. Table 2 lists the results of each sample set including the average percentage of cohesive failure within the adhesive versus the adhesive failure fraction on the contaminated and uncontaminated beams. The same results are shown graphically in Figures 5 and 6. The results show that, due to the high scatter and small number of the control samples, there are no statistically significant conclusions. The trend, however does show that higher pressure, more passes, larger amounts of silicone on the original cloth (threaded areas) do lead to decreased fracture energy and increased adhesive failure at the contaminated beam. Additional testing with a greater number of control samples would likely reduce the high CV and lead to statistically significant conclusions. In addition, separate chemical testing of TDCBs contaminated using the procedure outlined in the experimental section showed that 3 passes, high pressure, and threaded areas (most severe test conditions) did succeed in transferring a detectable amount of silicone to the grit blasted surface.

Sample set	Pressure used for exposure	Number of passes used for exposure	Type of cloth for exposure
1	Low	1	Clean
2	Low	3	Clean
3	High	1	Clean
4	High	3	Clean
5	Low	1	Contaminated
6	Low	3	Contaminated
7	High	1	Contaminated
8	High	3	Contaminated
9	Low	1	Threaded areas
10	Low	3	Threaded areas
11	High	1	Threaded areas
12	High	3	Threaded areas
13	Uncontaminated Control		

Table 1. Sample Set Descriptions

Sample set	Fracture energy	% Cohesive failure w/in adhesive	% Adhesive failure at contaminated beam; % at uncontaminated beam
1	102% of control samples, CV=3%	Avg =90, CV=0%	Avg =7, CV=25%; Avg =3, CV=58%
2	99% of control samples, CV=2%	Avg =92, CV=3%	Avg =6, CV=60%; Avg =2, CV=108%
3	92% of control samples, CV=6%	Avg =92, CV=0%	Avg =6, CV=37%; Avg =2, CV=89%
4	94% of control samples, CV=4%	Avg =81, CV=17%	Avg =10, CV=72%; Avg =9, CV=73%
5	99% of control samples, CV=6%	Avg =83, CV=7%	Avg =11, CV=54%; Avg =5, CV=78%
6	84% of control samples, CV=1%	Avg =78, CV=15%	Avg =20, CV=69%; Avg =2, CV=173%
7	100% of control samples, CV=5%	Avg =87, CV=9%	Avg =9, CV=99%; Avg =4, CV=96%
8	66% of control samples, CV=50%	Avg =62, CV=12%	Avg =36, CV=23%; Avg =2, CV=25%
9	94% of control samples, CV=2%	Avg =87, CV=3%	Avg =13, CV=20%; Avg =0, CV=173%
10	90% of control samples, CV=4%	Avg =72, CV=4%	Avg =22, CV=18%; Avg =3, CV=94%
11	92% of control samples, CV=6%	Avg =77, CV=8%	Avg =17, CV=33%; Avg =6, CV=0%
12	70% of control samples, CV=9%	Avg =27, CV=29%	Avg =73, CV=10%; Avg =0, CV=0%
13	Control Sample, CV=12%	Avg =77, CV=4%	Avg =0, CV=0%; Avg =23, CV=12%

Table 2. Sample Set Results

Conclusions

The results of the transfer testing show that the silicone was successfully transferred to the TDCBs under the most extreme test conditions. This was confirmed with both mechanical and chemical testing. This shows that the test method is capable of discerning the effect of contaminate transfer and can be used as an evaluation test method in future cases of contaminated materials.

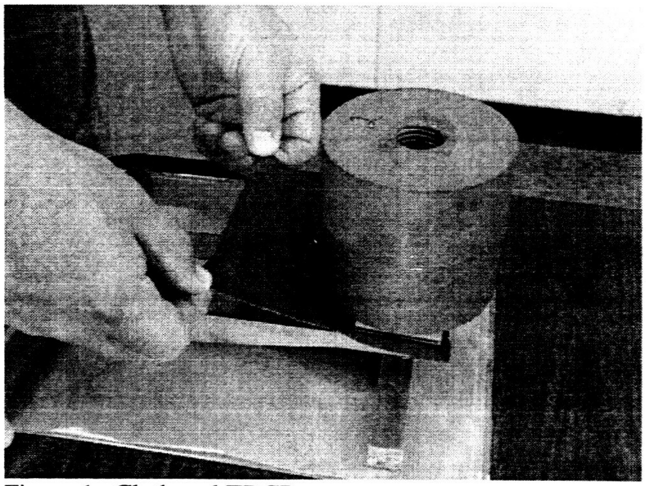

Figure 1. Cloth and TDCB exposure

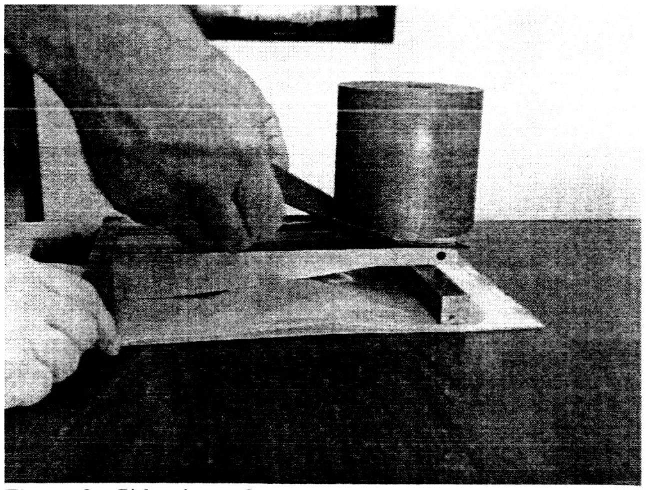

Figure 2. Side view of same

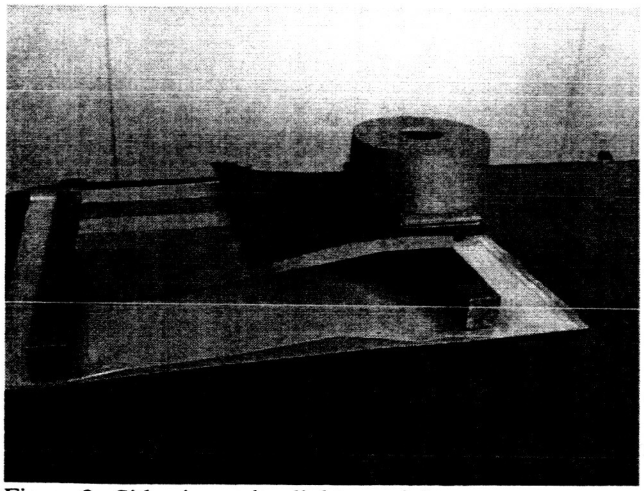

Figure 3. Side view using lighter weight

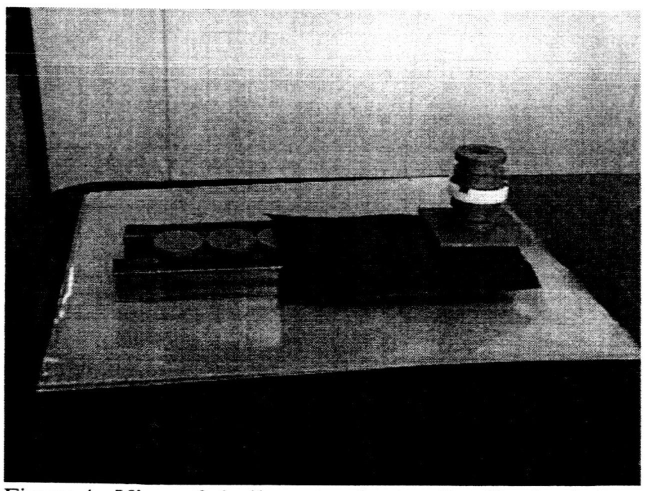

Figure 4. View of similar setup for tensile adhesion buttons

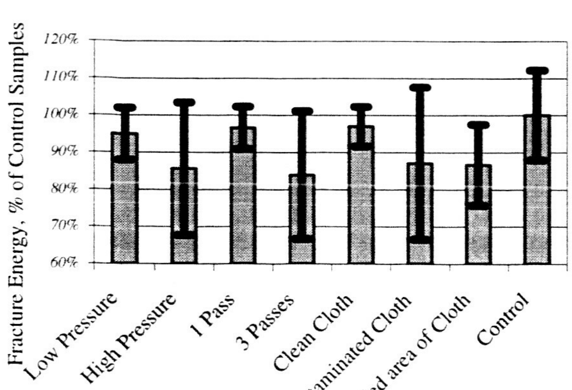

Figure 5. Categorized Averages of fracture energy reported as a percent of Control samples with Standard Deviation

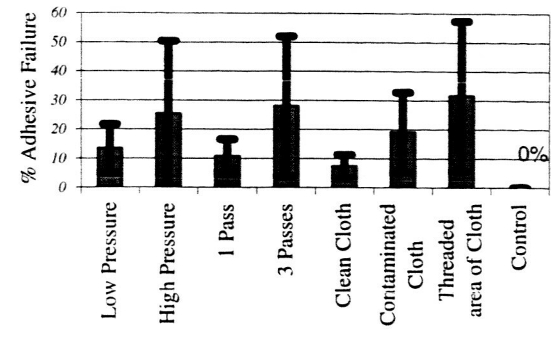

Figure 6. Average % Adhesive Failure at Contaminated Beam with Standard Deviation